National High Magnetic Field Laboratory
Operated by Florida State University, University of Florida, Los Alamos National Laboratory
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Here is a list of questions and comments that should help users' have the best chance for success with their high pressure experiments. Each user should take time to read over the list and respond to any that concern his/her particular experiment. Any additional comments are always welcome.

Please provide a list of citations (a review article would be ideal) of literature that pertains to this research. Please send any available optical or electrical ambient and high pressure data for this sample or a similar system. These items and the samples should be sent to Stan Tozer at least two months before the experiment is to be performed.

For communications prior to your arrival, is email, phone, or FAX preferred?

Sample Preparation and Handling:

The sample must be thinner than 40 um for optical work and 30 um for electrical work. Experiments above 20 GPa require even thinner samples. Typically, thinning the sample to these dimensions is done by cleaving or lapping. The lapping process entails mounting the sample(s) onto a brass chuck with a hot wax. The sample is then ground down to approximately 60 um using oil and diamond impregnated plates. The sample is further thinned and polished to the final thickness and a 1/10 um finish using diamond paste. In using this mounting wax, the sample will be exposed to temperature between 150 and 200 C for a period of 3-10 minutes. If this process will damage the sample, a pine resin can be used in place of the wax. Would the sample be contaminated by direct contact with the brass plug, the wax or pine resin, the diamond paste, or the lapping oil? For semiconductors grown on GaAs substrates, a final 10-20 second etch is done with a room temperature solution of 0.5-1% Bromine in methanol to remove any residual damage from the lapping process. If there is a more appropriate etch, please specify the etchant, etch rates at various temperatures, required time of exposure, temperature of bath and stopper solution. Samples 1 x 1 mm2 in area will be required for lapping. Please send a sample at least four times this size. Samples taken from the edge of a wafer are not suitable for thinning. Please state the best method for cleaving or dicing the sample.

Is the sample(s) susceptible to humidity or static charge?

How fragile/soft/brittle is the sample? Will sonicating the sample cause damage? Are there any special handling requirements?

Please explain any hazardous properties associated with the sample. Is it toxic, flammable, radioactive, etc.?

If the sample is radioactive, please specify the number of microcuries.

Please state the exact sample formula, buffer layers, and any substrate. Please specify whether the sample is bulk, ceramic, single crystal, or thin film. If it is on a substrate, please scratch an "x" on the substrate side and specify all means by which the substrate can be removed.

Will any of the following chemicals or gases damage the sample?

___ethanol
___methanol
___methylene chloride
___ethylene chloride
___tetrachloride
___acetone
___isopropyl alcohol
___water (hot, cold, and steam)
___Toluene (GE varnish solvent)
___N,N-Dimethyacetamide (used to dissolve conductive epoxy; requires boiling in this solution at approximately 120 C for 15 minutes)
___N,N-Dimethyl Formamide
___formic acid
___nitric acid
___sulfuric acid
___hydrochloric acid
___butyl acetate
___Acetic Ester (conductive paint solvent)
___Decyl Acetate (conductive paint solvent)
___Octyl Acetate (conductive paint solvent)
___Ethylene Glycol Mono Ethyl Ether Acetate (conductive paint solvent)

These answers will dictate the sample volume, anvil tip size, and pressure fluid.

What maximum pressure is required for the experiment?

Is the sample susceptible to strain or stress?

One of the following will be used as a pressure fluid. Which ones **must not** be used?

____ glycerin
____ helium
____ argon
____ nitrogen
____ xenon
____ 3M FC77
____ 3M FC70
____ methanol:ethanol:water::16:3:1
____ methanol:ethanol::4:1

Specific questions/comments for optical work:

Please specify the required excitation wavelength and maximum permissible laser power density.

Please indicate the ambient pressure signal level and wavelength as this dictates the type of anvil to be used -- type I or II diamond or sapphire.

Ruby is used as a pressure manometer.
Will any spectral features of the sample be obscured by the ruby doublet?

The ambient pressure, room temperature values of R1 and R2 are 694.2 and 692.8 nm, respectively, and the value of R1 is 693.4 at 4.2 K. The lines shift 0.365 nm/GPa into the red at all temperatures. Do you expect these lines to interfere with your experiments? We have some alternative ways to measure pressure.

Because the final injection point for the excitation source is 1.5 mm (the typical thickness of a diamond) from the sample, the light emitted from the end of a fiber optic spreads significantly and illuminates the entire sample and any exposed substrate edges. Collection of the sample signal is also not as efficient as that obtained with a lens system. Can a fiber optic be used to bring the excitation and signal to and from the sample or will direct focusing by lenses be required? A lens system is far more time consuming to implement and use.

Will ambient pressure data be required?

If so, can the fiber be directly attached to the sample with GE varnish (there will be no varnish between the sample and fiber end)? If not, the sample will be placed in a cup with a small gap between it and the fiber end.

Is the sample isotropic? If not, please supply a sketch showing the sample, the required field direction, and the light path.

Specific questions/comments for electrical work:

Please describe all the ways contacts have been made to this material and tell which ones worked best. For the described contacts, please list the ohmic behavior and the Ohm-square cm achieved. We typically perform a 30 to 60 second low energy argon sputter etch followed by sputtered contacts of gold (1000Å) defined by a metal mask to improve the contact resistance. This method will be used unless it will damage the sample.

Contacts are sometimes made with indium or indium alloy solder or low temperature cure epoxies. What is the maximum temperature to which the sample can be subjected and for what period of time? Will an inert gas or vacuum environment be required while the temperature is elevated?

Contacts can sometimes be enhanced by passing 1 to 3 second pulses of 0.5 to 3 mA of current. Will this damage the sample?

How do you remove surface oxides from the sample? How rapidly does the oxide regenerate after exposure to air?

Is the sample isotropic? If not, please supply a sketch showing the sample with its contacts and the required field direction.

Is the sample photoconductive? A fiber optic system without lenses is used to bring 488 nm laser light to the ruby chip used for pressure calibration. Will this light affect the sample? If so, how?

One or more of the following epoxies/paints may be used in the sample prep. Are there any problems with the resins or the metals used in them with regard to this sample?

3M #8 epoxy
3M #9 epoxy
3M #10 epoxy
Epotek H20E
Crest 4954
3M DP 460
Duco cement
5-minute epoxy
Master Bond EP 21TDC
Tra-Con FS209
Tamura L-100 gold paint
GE-7031 Varnish
DuPont 4929 Silver paint

Does contact between any of the following metals and the sample at temperatures between ambient and 70 C cause problems?

Indium
indium alloys
gold
silver
bismuth
gallium
nickel
lead
tin
copper
beryllium
mercury
cadmium

Experiment Planning - Pressures, Fields, and Temperatures:

The resistive magnets are scheduled in 3.5 hour shifts, four shifts per day, with most users getting two shifts per day. A typical magnet sweep from zero to maximum field to zero takes about 6 to 20 minutes. Users' must take as much data as possible while they have magnet time and plan on changing samples and doing other time consuming preparation when their power is off. The superconducting magnets are scheduled by the week. A magnet sweep from zero to maximum field to zero takes about 45 minutes. Superconducting magnets are available 24 hours per day, but sample changes and other things that require help from NHMFL staff members must be scheduled when the staff members are at work. The questions and information below will help users plan their experiments realistically.

What maximum pressure do you want?

What is the lowest pressure you want?

If ambient pressure data is required, do you want to use the small piece of sample in the high pressure cell with no pressure fluid, or can you use a different, larger piece? Changing and equilibration of the pressure takes 30-90 minutes. Warming the sample from 1.2 K to room temperature, changing the pressure, and cooling it to 4.2 K takes 2-3 hours. Each magnet shift is 3.5 or 7 hours long. You can do between 4 and 15 pressure points in a week. How many pressure points do you want to do?

The pressure in the cell changes with temperature. If there is a pressure induced phase transition near the maximum pressure desired, the pressure can be changed at the temperature of interest. The risk associated with this procedure is that the pressure may not be hydrostatic.

Measurements can be done between 25 mK and 600 K. Each temperature point takes between 20 and 60 minutes, sometimes longer. The time depends on the thermal characteristics of the sample holder, how the temperature is controlled, and the number and speed of magnet sweeps at each temperature.

Please list the temperature points to be used with each sample.

How much will the sample signal change with a 1% (10%) variation in temperature?

Does temperature cycling damage the sample? Does a rapid temperature change damage the sample? If so, how fast do you want to change the sample temperature?

Will it be necessary to keep the sample cold/hot for several days once the experiment has started?

Do you want to cool (or heat) the sample very slowly with the magnetic field on? If so, please describe the process and tell how high the field must be.

What is the maximum value of field (DC and pulse) that you need?